



# Green Synthesis and Characterization of ZnO Nanoparticles using *Hibiscus rosa-sinensis* Leaf Extract

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## ABSTRACT

The synthesis of nanoparticles was investigated, for their exhibit in larger surface area, thus opening too much potential with respect to all technological applications. In the present study, ZnO nanoparticle is prominent in scientific fields since it combines knowledge from physics, chemistry, biology, medicine, informatics, and engineering, etc. Nanoparticles produced by plants are more stable. The synthesized nanoparticles were characterized by using various techniques by XRD, SEM, EDAX, FTIR, and Antibacterial Activities. The synthesized nanoparticle was formed by using zinc nitrate doped with *Hibiscus Rosasinensis* leaf extract. The Diffraction (XRD), Scanning Electron Microscope (SEM), Electron dispersive X-ray analysis (EDAX), Fourier transform infrared spectroscopy (FTIR).

**Keywords:** *Hibiscus rosa-sinensis*; Scanning electron microscopy; Zinc Oxide.

## 1. INTRODUCTION

Nanotechnology is dedicated to providing clear and concise explanations of nanotechnology applications. Scan the listings below to find an application of interest, or use the navigation bar above to go directly to the page discussing an application of interest (Chaudhary *et al.* 2019).

Green synthesis is an emerging area in bionanotechnology and provides economic and environmental benefits as an alternative to chemical and physical methods. In this method, nontoxic, safe reagents which are eco-friendly and safe are used. Various natural resources available in nature, such as plant extracts, cyclodextrin, chitosan, and many more, have been studied to synthesize metal oxide nanoparticles (Devi *et al.* 2015).

Zinc (Zn), a chemical element, and a low-melting metal of Group 12 (IIB, or zinc group) of the periodic table, is essential to life and is one of the most widely used metals. Zinc is of considerable commercial importance. Zinc is an important trace element in the human body. It is found in high concentrations in the red blood cells as an essential part of the enzyme carbonic anhydrase, which promotes many reactions relating to carbon dioxide metabolism. The zinc present in the pancreas may aid in the storage of insulin. Zinc is a component of some enzymes that digest protein in the gastrointestinal tract. Zinc deficiency in nut-bearing and fruit trees causes such diseases as pecan rosette, little leaf, and mottle leaf. Functions of Zinc in the

hemocytoblasts of snails' blood to transport oxygen in a way of analogous to iron in the human blood (Diallo *et al.* 2015; Hong *et al.* 2006).

Zinc oxide is a white, powdery mineral with a long history of use as sun protection. It is also used to create other products, such as diaper rash ointments and makeup. ZnO nanoparticles easily dissolve in soil and are uptaken by plants. They are employed in a wide range of applications in agriculture due to their unique properties. Results suggest that the application of ZnO NPs could increase plant growth and development (Hong *et al.* 2009; Manokari and Shekhawat, 2017).

Zinc nitrate does not have a broad scale use but is used for the synthesis of coordinating polymers on a laboratory scale. The controlled decomposition of zinc oxide can also be used for the generation of various structures, including nanowires. It can also be used as a dyeing mordant. Zn (NO<sub>3</sub>)<sub>2</sub> is an inorganic chemical compound with the chemical name Zinc nitrate. It is also called Zinc dinitrate or Celloxan, or Zinc Nitrate Hexahydrate. It is widely used as a catalyst to manufacture medicine, dyes, and various other chemicals. Inhaling dust causes irritation in the throat and nose. Swallowing Zinc dinitrate can lead to corrosion of the alimentary tract. Contact with the skin results in irritation and can cause rashes (Pal *et al.* 2019; Sirelkhatim *et al.* 2015).

A natural dye has been tried with the *Hibiscus rosa-sinensis* flowers and leaves extract on cotton fabric. Can use the flowers and leaves of the hibiscus to produce

a lovely reddish Hibiscus dye. The following methods such as Ultrasonic Automiser and Padding Mangle were used for dyeing. Treated sample has moderate to fair colorfastness properties. The physical and mechanical properties of the treated samples seemed to be good (Sridar *et al.* 2018). In the present study, Hibiscus rosa-sinensis leaf extract (HLE) can act as a natural coagulant for the water treatment was tested. An insignificant effect of alkalinity on the performance of HLE was observed. The addition of NaCl increased the dissolution of active coagulation species and enhanced the efficiency of HLE significantly. But the optimal dosages of HLE were lesser than that of alum. Thus, HLE can be used as a coagulant aid for the effective treatment of water (Vidya *et al.* 2013).

## 2. MATERIALS & METHODS

### 2.1 Material

All the chemical such as Zinc nitrate, distilled water, and other sodium hydroxide ingredients utilized in this work purchased from Erode, Tamilnadu. The leaves of Hibiscus rosa-sinensis leaf collected from in and around Arachalur, Tamilnadu, India.

### 2.2 Preparation of Hibiscus rosa-sinensis Leaves Extract

The plant extract was prepared by taking 20g of Hibiscus rosa-sinensis leaves. The leaves were washed several times using running tap water and then again washed double distilled water to remove dust particles, then dried to boiled 25 minutes in 100ml of distilled water. Then the solution is changed to a light green color, and the extract was filtered and stored at room temperature.

### 2.3 Green Synthesis of ZnO and Leaf Capped Nanoparticles

Hibiscus *rosa-sinensis* Leaf was collected from the surrounding area and washed several times using running tap water and then again washed double distilled water to remove dust particle then dried to remove residual Moisture. The plant extract was prepared by taking 10g of Hibiscus *rosa-sinensis* leaves and boiled the leaves for 25 minutes in 100ml of distilled water. Boiled them for 30 minutes, and the extract was filtered using Whatman filter paper to get a clear solution. In this method, 10g of Zinc nitrate was dissolved with 100ml of distilled water and stirred for about 30 minutes. After that, the leaf extract of 10ml drop-wise into the above solution and changed the color of the solution to light green color, and sodium hydroxide solution was added drop-wise to the mixture to maintain PH level at 12. The

synthesized sample was aging at 24 hours. Thus the scatted residue was kept in a microwave oven at 350w for 30 minutes. The dried product was grained in a mortar, and then the fine leaf ZnO nanoparticles were obtained.



Fig. 1: Hibiscus rosa-sinensis Flower and Leafs

### 2.3 Characterization Techniques

#### 2.3.1 XRD Analysis

The prepared samples were analyzed using XRD (X – ray Diffraction) technique. This XRD pattern predicts the lattice parameter (a and c), unit cell volume, and crystalline size of the sample. The XRD pattern of prepared samples was well-matched with JCPDS File. The lattice parameter of the sample was calculated using the following equation:

$$1/d^2 = (4(h^2+hk+k^2)/3a^2) + (1^2/c^2))$$

Where d is the spacing between the planes, a and c are the lattice parameter. The unit cell volume (V) of the sample was described using the given equation:

$$V = (\sqrt{3}/2) + a^2 + c^2$$

The average crystalline size of the sample was determined by using Scherer's formula.

$$D = K\lambda/\beta \cos \theta$$

Where D denotes the average crystalline size of the sample, K represents the broadening constant,  $\lambda$  denotes the wavelength of CuK $\alpha$  radiation source (1.54Å),  $\beta$  represents the full width at half maximum, and angle of diffraction is denoted by  $\theta$ .

### 2.3.2 FTIR Analysis

FTIR (Fourier transform spectroscopy analysis) is an analytical methodology used in industry and academic laboratories to understand the structure of individual molecules and the composition of molecular mixtures.

### 2.3.3 SEM & EDAX Analysis

Scanning electron microscopy with energy-dispersive X-ray spectroscopy (SEM/EDX) is the best known and most widely used surface analytical technique. High-resolution images of surface topography, with excellent depth of field, are produced using a highly focused, scanning (primary) electron beam.

## 3. RESULTS & DISCUSSION

### 3.1 XRD Analysis

XRD analysis is used to determine the crystalline size and phase identification of the nanoparticles. The XRD pattern of ZnO nanoparticles is shown in Fig 1.2. The XRD pattern indicates that the ZnO sample is a hexagonal structure, and it well-matched with JCPDS files. For ZnO the average crystalline size was nm was shown in (Table 1). The diffraction peaks of ZnO nanoparticles at  $2\theta = 36.44$ ,  $47.71$ , and  $69.290$  which having planes of ZnO for 101, 102, and 201, respectively. Then calculated by using the Debye-Scherrer formula,

$$D = k \lambda / \beta \cos \theta$$

Where D is the crystallite size, k is the Scherrer constant,  $\lambda$  is the wavelength of X-Ray source (Å),  $\beta$  is

the full width at half maximum of the diffraction peak, and  $\theta$  is the Bragg's angle.

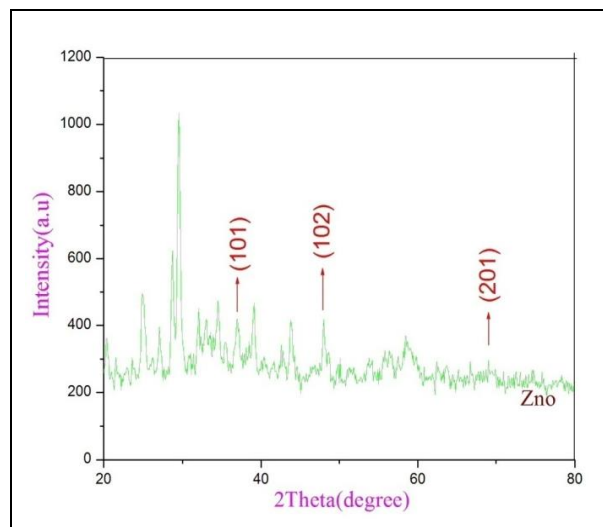


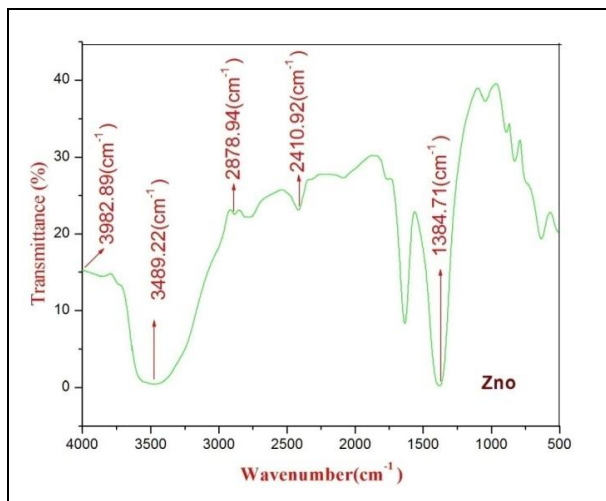
Fig. 2: Synthesized Sample with XRD Pattern for ZnO

### 3.2 FTIR Analysis

FTIR spectra of the prepared ZnO samples were recognized using a wavelength range of  $400 - 4000 \text{ cm}^{-1}$  is shown in Table 2. The observed peak resulted from the green synthesis method is at  $3861.49$  to  $879.54 \text{ cm}^{-1}$ , whereas from the green synthesis method, the peaks observed at  $3960.65$  to  $570.92 \text{ cm}^{-1}$ . The vibrations of a variety of groups are present at different wavenumbers of IR radiation. The broad peak was absorbed at  $3861.49 \text{ cm}^{-1}$  and  $3960.65 \text{ cm}^{-1}$  (Alcohol), which contact to O-H stretching band. C-H stretching confirms from the absorption peak of  $2800.64 \text{ cm}^{-1}$  and  $2809.44 \text{ cm}^{-1}$  (Alkynes). N=O stretching from the absorption the peaks at  $1450.47 \text{ cm}^{-1}$  and  $1450 \text{ cm}^{-1}$  (Nitro). The FTIR spectrum absorbs the peak at  $3589.53 \text{ cm}^{-1}$  and  $3446.81 \text{ cm}^{-1}$  were calculated with the stretching vibrations of N-H (Amine) bond. Introducing a capping agent has created a minor change in the functional group analysis of the samples. The spectrum (Fig. 3) reveals the FTIR graph of ZnO.

Table 1. Lattice Constant, Crystalline Size, and Unit Cell Volume of the Synthesized Samples.

Sample name	2 $\theta$ (deg)	FWHM (deg)	D (Å)	Intensity (Counts)	Crystalline Size (nm)	Average Crystalline Size (nm)	hkl	Lattice Constant		UnitCell Volume (Å <sup>3</sup> )
								a=b	c	
ZnO	36.44	0.47	2.46	119	17.79	18.47	101	3.1	5.2	45.60
	47.71	0.49	1.90	283	17.72		102			45.82
	69.29	0.48	1.35	163	19.92		201			47.81



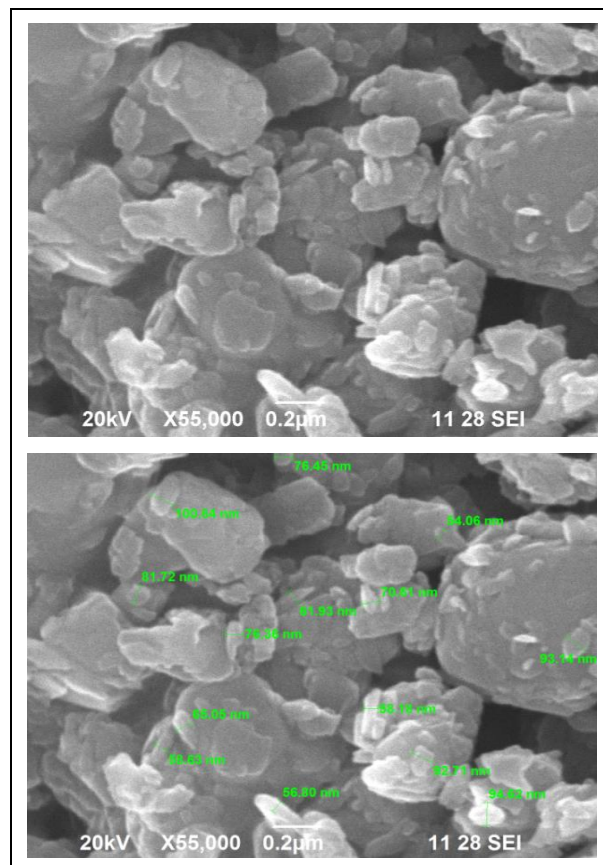
**Fig. 3: FTIR spectrum of ZnO**

### 3.3 SEM and EDAX

The Scanning electron microscope (SEM) analysis determines the shape and Morphology of ZnO nanoparticles. Fig. 4 illustrated the morphological descriptions and elemental composition of ZnO, shows Spherical shaped morphology for ZnO. The capping agent can create a minor change in the morphology of the sample.

The Energy Dispersive X-Ray Spectroscopy is used to investigate the elemental composition and chemical analysis of ZnO. The EDAX analyses consist of spectra showing peaks corresponding to the elements making up the accurate composition of the sample.

The analysis observed Zn (Zinc), O (Oxygen) for ZnO. It represents the purity of the sample. In, EDAX the presence of Zn and O reveals the absence of impurities in the sample. The EDAX analysis has shown in Fig. 5.



**Fig. 4: SEM Analysis of ZnO**

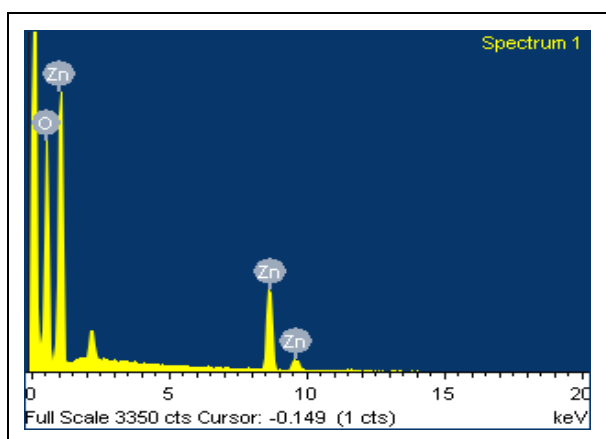
**Table 2. Functional group of ZnO**

Sample Name	Wave Number( $\text{cm}^{-1}$ )				
	O-H Stretching vibration (free)	O-H Stretching vibration (banded)	C-H Stretching vibration	N=O Stretching vibration	N-H Stretching vibration
ZnO	2410.92	3982.89	2878.94	1384.71	3489.22



**Table 3. EDAX Spectra of Pure ZnO**

Sample	Element	App Conc.	Intensity Corr'n	Weight%	Weight% Sigma	Atomic%
ZnO	O	43.77	1.3608	47.92	0.69	78.99
	Zn	30.47	0.8718	52.08	0.69	21.01
Totals			100.00			

**Fig. 5: EDAX analysis of ZnO**

#### 4. CONCLUSION

In this present study, the synthesis of Zinc oxide nanoparticles by the green synthesis method was reported. XRD analysis predicts the crystalline size, lattice parameter, and unit cell volume of the sample. The average crystalline size is 18.47nm. From FTIR study reveals the functional groups present in the sample. SEM analysis revealed the morphological structure, and it shows spherical-shaped morphology. EDAX analysis determines the sample's elemental composition, such as Zn (Zinc), O (Oxygen) for ZnO. It represents the purity of the product.

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